

## ASTM standards for fire debris analysis: a review

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### Abstract

The American Society for Testing and Materials (ASTM) recently updated its standards E 1387 and E 1618 for the analysis of fire debris. The changes in the classification of ignitable liquids are presented in this review. Furthermore, a new standard on extraction of fire debris with solid phase microextraction (SPME) was released. Advantages and drawbacks of this technique are presented and discussed. Also, the standard on cleanup by acid stripping has not been reapproved.

Fire debris analysts that use the standards should be aware of these changes.

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### 1. Introduction

The American Society for Testing and Materials (ASTM) has provided standards for fire debris analysis since 1990. Recently, important changes have been made in the two main standards for the analysis of ignitable liquid residues, ASTM E 1387 and ASTM E 1618 for gas chromatography (GC) and gas chromatography–mass spectrometry (GC–MS), respectively. Furthermore, one standard has not been reapproved since its adoption in 1995 and one has been added. The purpose of this communication is to review the available standards for fire debris analysis and to present the changes in the two main analytical standards. It is essential that fire debris analysts following ASTM standards update their library and be aware of these changes.

### 2. Unchanged standards

The following standards remain unchanged since the last edition:

ASTM E 1385-00 Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Steam Distillation [1].

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ASTM E 1386-00 Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris by Solvent Extraction [2].

ASTM E 1388-00 Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples [3].

ASTM E 1412-00 Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal [4].

ASTM E 1413-00 Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration [5].

### 3. New standard

The following standard was published in the 2002 Annual Book of ASTM Standards:

ASTM E 2154-01 Standard Practice for the Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME) [6].

SPME was developed in 1989 and is applied to a wide range of different kinds of analysis [7–10]. SPME method has been shown to present an interesting sensitivity and rapidity [11,12]. In addition, it is a completely non-destructive technique. However, a few disadvantages need to be addressed.

First, the fiber possesses a very limited number of adsorption sites, and an important displacement might occur, even at low concentrations. Ren and Bertsch [13] showed that the selectivity bias of the SPME method can be avoided by using two kinds of fibers, which of course requires two analyses per sample. The ASTM standard makes the user well aware of the problem under sections 5.4, 6.4.1, and 6.4.2. The latter states, “Temperatures above 80 °C may result in a significant discrimination against high volatility compounds when the 100 µm PDMS fiber is used”.

Second, there is no real automation in the task and the analyst is forced to perform all the steps manually. Other methods, such as passive headspace concentration on activated charcoal (ASTM E 1412-00), might present a total longer procedure, but the time for which the analyst is required to pay attention is actually quite brief.

Third, the use of the same fiber for every sample requires that the “blank” fiber (after cleaning) be injected and observed. This step doubles the length of analysis time. Some other methods do not necessarily require the use of a blank between each sample, because the same separation device is not used over. Again, the ASTM standard addresses this limitation under sections 6.10, 6.10.1 and 7.1.

Often, a new technique is perceived by the scientific community as better than the old one(s); however, this is not always true. While SPME offers an interesting sensitivity and speed, it is no better than other existing extraction methods, such as passive headspace concentration with activated charcoal. Fire debris analysts are strongly advised to read the existing literature on SPME and form their own opinion before adopting this new method. Furthermore, the ASTM standard does not define this method as suitable for being the sole extraction method. Section 4.5 states, “This practice is intended for use in conjunction with other extraction techniques described in Practices E 1385, E 1386, E 1412, and E 1413”.

#### 4. Standard not reapproved

The following standard has not been reapproved in 2002 due to a low success rate and consequent infrequency of use:

ASTM 1389-95 Standard Practice for Cleanup of Fire Debris Sample Extracts by Acid Stripping [14].

Acid stripping is a procedure used to “clean” extracts from oxygenated or nitrogenated organic compounds. This was applied when co-eluting interfering peaks were present in the chromatogram. However, the increasing popularity of mass spectrometry as a detection technique after the chromatographic separation and the use of selected ion monitoring offer a better means of eliminating these interferences without the cumbersome use of acids. An extensive literature treating that topic can be found since 1982 [15–20].

ASTM policy is to withdraw any standard that has not been reviewed or reapproved in the 5 years following its

adoption or last revision. It is expected that this standard will be withdrawn next year.

#### 5. Revised standards

The following standards underwent important changes in 2001:

ASTM E 1387-01 Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography [21].

ASTM E 1618-01 Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry [22].

It is also interesting to note that ASTM E 1618 is no longer a standard guide, but a standard test method. This means that the method is no longer directly related to E 1387, but serves as a “stand alone” method. To this effect, paragraph 1.3 has been changed.

The main change introduced in the revised versions of the ASTM E 1387 and E 1618 standards is the classification system of the ignitable liquids. The earlier classes had both numbers and names as shown in Table 1. With the evolution of the petroleum industry, new products were developed and the old classification system had to adapt to these new products. On this topic, DeHaan [23] wrote an excellent description of the changes that occurred in the last several years. As an example, more and more products were classified in the “0 miscellaneous” category and the number of subcategories of this class exceeded the total number of classes.

The new system is a two-dimensional classification as presented in Table 2. More categories have been defined, and each category is divided in three subcategories “light, medium and heavy”, with the exception of the gasoline category. “Light” means a carbon range from C<sub>4</sub> to C<sub>9</sub>, “medium” from C<sub>8</sub> to C<sub>13</sub>, and “heavy” from C<sub>8</sub> to C<sub>20</sub> and above.

Criteria to interpret and identify ignitable liquid residues are not as specific in E 1387 as they are in E 1618, since the latter includes mass spectral characteristics. It is not the

Table 1  
Previous ASTM classification system of ignitable liquids

Class number	Class name
1	Light petroleum distillates (LPD)
2	Gasoline
3	Medium petroleum distillates (MPD)
4	Kerosene
5	Heavy petroleum distillates (HPD)
0	Miscellaneous
0.1	Oxygenated solvents
0.2	Isoparaffins
0.3	Normal alkanes
0.4	Aromatic solvents
0.5	Naphthenic/paraffinic solvents

Table 2  
New ASTM classification system

Class	Light	Medium	Heavy
Gasoline		Fresh gasoline typically falls in the range of C <sub>4</sub> –C <sub>12</sub>	
Petroleum distillates	Petroleum ether, some cigarette lighter fluids, some camping fuels	Some charcoal starters, some paint thinners, some dry cleaning solvents	Kerosene, diesel fuel, some jet fuels, some charcoal starters
Isoparaffinic products	Aviation gas, specialty solvents	Some charcoal starters, some paint thinners, some copier toners	Some commercial specialty solvents
Aromatic products	Some paint and varnish removers, some automotive parts cleaners, xylenes, toluene-based products	Some automotive parts cleaners, specialty cleaning solvents, some insecticide vehicles, fuel additives	Some insecticide vehicles, industrial cleaning solvents
Naphthenic-paraffinic products	Cyclohexane based solvents/products	Some charcoal starters, some insecticide vehicles, some lamp oils	Some insecticide vehicles, some lamp oils, industrial solvents
<i>n</i> -Alkanes products	Solvents, pentane, hexane, heptane	Same candle oils, copier toners	Some candle oils, carbonless forms, copier toners
De-aromatized distillates	Some camping fuels	Some charcoal starters, some paint thinners	Some charcoal starters, odorless kerosene
Oxygenated Solvents	Alcohols, ketones, some lacquer thinners, fuel additives, surface preparation solvents	Some lacquer thinners, some industrial solvents, metal cleaners/gloss removers	
Others-miscellaneous	Single component products, some blended products, some enamel reducers	Turpentine products, some blended products, various specialty products	Some blended products, various specialty products

intent of this paper to review the criteria for E 1387, since GC alone is less and less used in the laboratory and should, in the opinion of the authors, be abandoned. However, it is interesting to note the statement made in section 11.6 of method E 1618, “All the components necessary for an identification by Practice E 1387 must be present in order for an identification to be made by this test method”.

According to E 1618, the categories are defined as follows:

The *gasoline* category, as defined in section 10.3.1 represents “All brands of gasoline including gasohol”. Gasoline is usually characterized by the abundant and specific pattern of aromatics with a low level of aliphatics.

The *distillates* category is defined in section 10.4. It includes the traditional petroleum distillates and should present the Gaussian distribution pattern. Aromatic compounds are present.

*Isoparaffinic products* are defined in section 10.5. This category includes products that are composed exclusively of branched aliphatics. *n*-Alkanes, cycloalkanes and aromatics are usually absent or present at insignificant levels.

*Aromatic products* are defined in section 10.6. They include all products that contain almost exclusively aromatic (including polyaromatic hydrocarbons) compounds.

The *naphthenic-paraffinic products* category is defined in section 10.7. It includes all products that contain mainly branched aliphatics and cycloalkanes. *n*-Alkanes and aromatics are usually absent or present at very low levels.

This category is distinguished from the isoparaffinic products category by the predominance of the cycloalkanes.

*Normal alkane products* are defined in section 10.8. They include all products that present exclusively *n*-alkanes. Isoparaffinics, cycloalkanes, and aromatics are absent.

*De-aromatized distillates* are defined in section 10.9. It includes petroleum distillates that do not show the presence of aromatics, or an unusually low concentration of aromatics.

*Oxygenated solvents* are defined under section 10.10. These consist of products that contain major oxygenated components. This is usually easily observed through the tailing present in the chromatograms, particularly with the early eluting peaks.

A *miscellaneous/other* category is presented in section 10.11, however, most of the encountered products should fit in one of the previous categories.

[Table 3](#) presents a summary of the main classes with their characteristics. This should help the fire debris analyst in the choice of the proper class.

As defined in section 10.1.3.4, the frame “light, medium, heavy” is not rigid and “It may be necessary to characterize a product as “light to medium”, or “medium to heavy”, when the carbon number range does not fit neatly into one of the above categories”.

Extracted ion profiles are most useful when distinguishing between categories such as distillates, isoparaffinic products and naphthenic-paraffinic products. As an example, a quick overlay of extracted ions 55, 57, 83, and 105 should allow the analyst to quickly differentiate between petroleum distillates, naphthenic-paraffinic, isoparaffinic products, and de-aromatized distillates. Using GC alone, such distinctions are usually not possible.

Table 3  
Main ASTM classes and their characteristics

Class	Alkanes	Cycloalkanes	Aromatics	PNAs
Gasoline	Present, less abundant than aromatics	Present, less abundant than aromatics	Abundant	Present
Petroleum distillates	Abundant, Gaussian distribution	Present, less abundant than alkanes	Present, less abundant than alkanes	Present (depending on boiling range), less abundant than alkanes
Isoparaffinic products	Branched alkanes abundant, <i>n</i> -alkanes absent or strongly diminished	Absent	Absent	Absent
Aromatic products	Absent	Absent	Abundant	Abundant (depending on boiling range)
Naphthenic paraffinic products	Branched alkanes abundant, <i>n</i> -alkanes absent or strongly diminished	Abundant	Absent	Absent
<i>n</i> -Alkanes products	Abundant	Absent	Absent	Absent
De-aromatized distillates	Abundant, Gaussian distribution	Present, less abundant than alkanes	Absent or strongly diminished	Absent or strongly diminished
Oxygenated solvents	Composition may vary, presence of oxygenated organic compounds			

## 6. Conclusion

The American Society for Testing and Materials (ASTM) provides consensus standards for performing fire debris analysis. Recently, an important revision of the main analytical standards, E 1387 and E 1618, was effected. The new classification system includes more categories and provides for a more precise description of ignitable liquid residues. A solid phase microextraction standard has just been introduced; however, this method may present important disadvantages that need to be considered before widespread adoption ensues. Fire debris analysts should read these standards, be aware of the changes, and integrate these concepts into their reports.

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